Characterization of Single-Wall Carbon Nanotubes

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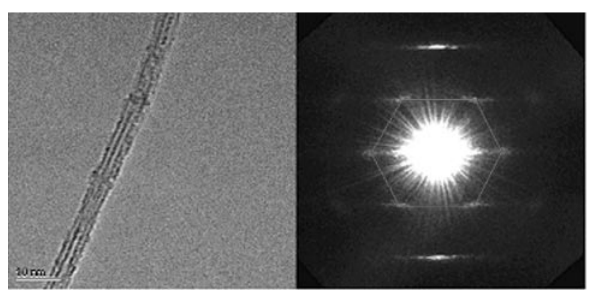
The Nanomaterials Characterization Team at the NASA Johnson Space Center (JSC) is a multidisciplinary effort to fully characterize the nanomaterials used for applications related to human spaceflight and exploration. Since 1997, this team has been focusing on characterizing unique features and properties of single-wall carbon nanotubes (SWCNTs). In 2003, the team developed a JSC characterization protocol to help standardize the assessment of the purity and dispersion of SWCNTs. The goals of the team are to explore new and novel techniques and to modify existing analytical techniques to characterize the raw, purified, and processed SWCNT materials.

The purpose of the characterization team is to provide vital feedback to the production, processing, and applications teams. Information provided by the team is used to monitor and evaluate the processes and progress within this are. The approach developed by the team is to characterize completely SWCNT material from the nanoscale to the macroscale by evaluating the physical and chemical properties of the material. The team uses analytical techniques that probe SWCNT materials over a wide range. The characterization of SWNCTs is aided through collaborations with commercial and academic institutions such as Rice University, the University of Illinois Urbana-Champaign, Smart Imaging Inc., and Ionwerks. The JSC Nanomaterials Characterization Team is working with the National Institute of Standards and Testing (NIST) and the Institute of Electrical and Electronic Engineers (IEEE) to establish SWCNT standards. In addition, as of 2003 JSC has been teamed with NIST to hold biennial workshops on SWCNT characterization to address critical characterization challenges associated with the purity and dispersion of SWCNTs. A result of these workshops is that in 2005 the team prepared a "practice guide for thermogravimetric analysis (TGA)," written as part of nanotube standards development in conjunction with both NIST and IEEE.

To evaluate comprehensively SWCNT materials, the JSC team has developed a characterization protocol that establishes numerical measures for homogeneity, dispersion stability, and impurity constituents. This protocol uses techniques such as scanning electronic microscopy (SEM) and transmission electron microscopy (TEM), energy dispersive x-ray analysis, Raman spectroscopy, TGA, and ultraviolet-visible-near infrared (UV-Vis-NIR) spectroscopy. In addition, the team also uses techniques such as x-ray photoelectron spectroscopy (XPS), near infrared (NIR) fluorescence, electron beam diffraction, inductively coupled plasmas (ICPs), high-pressure liquid chromatography, x-ray diffraction, and laser desorption ionization-time of flight-mass spectrometry (LDI-TOF-MS) to investigate the various properties of SWCNT material.

Quality control of SWCNTs grown through laser production is one of the major tasks of the team. Over the past several years the JSC Nanomaterials Characterization Team has observed that SWCNT material can display vast differences in its properties, depending on the parameters under which the material is produced or processed. In 2004, team members observed through characterization that the properties of



Nano area electron-diffraction pattern recorded from a bundle consisting of 3-4 individual nanotubes. This bundle consists of tubes of similar chiralities being "arm chair" or nearly arm chair nanotubes.

SWCNT material produced by the laser method vary according to the region of the production oven from which the material is harvested. Moreover, characterization has shown that the consistency of material produced from production run to production run shows both similarities and differences in its properties. In 2005, The JSC team began working with Rice University on a project called "Continued Growth of Nanotubes for Use in Quantum Conductors" that is aimed at making highly conducting nanotube cables using one particular type of nanotubes known as "arm-chair" nanotubes. Through use of techniques such as nano-area electron beam diffraction, Raman spectroscopy, UV-Vis-NIR absorption spectroscopy, and NIR fluorescence, the team is able to monitor and select these "arm-chair" nanotubes from the bulk SWCNT material.

Another task of the team is to determine the successful removal of impurities from as-produced materials through purification processes. Characterization enables the assessment of the degree purification. This assessment, in turn, provides feedback to the processing team of how the purification processes can be refined to enhance the removal of impurities. These purification processes incorporate the use of harsh chemical treatments that may result in modifications to the nanotube materials and are, therefore, closely monitored. One approach to purification involves annealing the material at various temperatures prior to acid treatment. Characterization of this material processing indicates that there is an optimal temperature at which the removal of metal impurities is enhanced. It has also been observed that, above this temperature, the SWCNT walls become damaged. In 2005, through use of XPS and ICP, the team was able to detect the presence of silicon in laser-produced SWCNT material, an artifact that results from the quartz oven use in production; data from XPS indicated that the silicon is present in the form of oxide and not as carbides. Also, a new characterization tool for the purity assessment of SWCNT is identified. The technique of LDI-TOF-MS is used to provide information on the fullerene content within both produced and purified SWCNTs (Figure 2). The fullerene trend line observed was compared to computational modeling and found to have good agreement. This helped confirm their identification as closed-shell fullerene structures.

To make SWNCT materials suitable for applications, nanotube materials must be processed or modified to enhance the property to meet the required needs of the application. Such processing can include the functionalization of the sidewalls of SWCNTs, deposition of metal particles into the nanotube matrix, isolation of type specific nanotubes through extractions, or dispersion of SWCNTs in solution. In this case, SWCNTs must be characterized to monitor not only the extent of the modification but also the effectiveness of the modification. The following is accomplished in 2005 to characterize the processed SWCNTs for applications: (1) XPS has been used to monitor the surface chemistry of SWCNT functionalized with amine-based side groups while TGA is used to determine the thermal stability; (2) XPS is also used to characterize the extent of platinum deposition in fuel cell membranes; (3) microtome sample preparation for TEM helped to examine the interface between the layers of the components of the electrodes for fuel cells and super capacitors; (4) Raman spectroscopy and SEM have been used to confirm the survival of SWCNTs embedded into aluminum metal through stir welding process; and (5) finally through the use of the SIMAGIS software, developed by Smart Imaging Inc., the dispersion of SWCNTs in solution is measured routinely with improved accuracy.